

SYNOPSIS

A series of mixed nickel-cadmium oxalates was prepared by co-precipitation. Nickel nitrate and cadmium nitrate solutions of 1 M were mixed in required proportions. The mixed solutions thus prepared were added dropwise to 1 M solution of oxalic acid with constant stirring. The resultant precipitate of mixed Ni-Cd oxalate was washed with hot distilled water and dried in an oven at loo^oC for 6 hours.

The thermal behaviour of these Ni-Cd oxalates was studied using the techniques such as (i) Thermogravime **Tric analysis** (TGA), (ii) Differential thermal analysis (DTA). The oxalates were also examined by X-ray diffraction method.

In thermogravimetric analysis nickel oxalate and co-precipitated mixed Ni-Cd oxalates give a single step for dehydration and for decomposition. The end product of thermal decomposition of $\text{NiC}_2\text{O}_4.2\text{H}_2\text{O}$ is NiO. The Ni-metal formed during the decomposition, in the initial stage oxidises to oxide. This is supported by the second exothermic peak in DTA studies.

Single peak in the DTA curves both for dehydration and decomposition supports the presence of homogeneous phase in the co-precipitated Ni-Cd oxalates. No linearity was observed for the relationship between the heat of reaction (Δ H) and percentage composition of cadmium for decomposition step. The values of Δ H for mixed oxalates are below the sum of individual components in the case of decomposition peaks. This indicates that the co-precipitated Ni-Cd oxalates are not physical.

mixtures but there is a close association of the components suggesting a homogeneous phase (or mixed crystals).

This view is supported by the observation of a single step for decomposition in the thermogram of the co-precipitated oxalates and two steps in thermogram for mechanically mixed Ni-Cd oxalates.

The oxides of Ni-oxalate, Cd-oxalate and their mixed oxalates were prepared by heating the oxalates in air at 800° C for 6 hours. The slight variation in 'd' values suggests that the mixed oxalates may be either mixed crystals or metastable solid solutions at the grain boundaries. The products of decombosition of mixed Ni-Cd oxalates in air medium are nickel oxide and cadmium oxide.

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